

REPORT DOCUMENTATION PAGE

AFRL-SR-AR-TR-03-

0360

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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE 04-AUG-2003	3. REPORT FINAL REPORT (01-JAN-1999 TO 31-JAN-2002)
4. TITLE AND SUBTITLE DETERMINATION OF STRAIN FIELDS AT THE NANOMETER SCALE WITH APPLICATIONS TO MEMS DESIGN AND MICROMECHANICS			5. FUNDING NUMBERS F49620-99-1-0091
6. AUTHOR(S) WOLFGANG G. KNAUSS IOANNIS CHASIOTIS			
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) CALIFORNIA INSTITUTE OF TECHNOLOGY MAIL STOP 213-6 PASADENA CA 91125			8. PERFORMING ORGANIZATION REPORT NUMBER
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) AFOSR/NA 4015 WILSON BOULEVARD ROOM 713 ARLINGTON VA 22203-1954			10. SPONSORING/MONITORING AGENCY REPORT NUMBER
11. SUPPLEMENTARY NOTES			
12a. DISTRIBUTION AVAILABILITY STATEMENT APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED			12b. DISTRIBUTION CODE
13. ABSTRACT (Maximum 200 words) The following summarizes progress on the program made in the period of August 1998 until September 1999. Several aspects of the material behavior and characteristic were addressed such as the material constants, the surface condition and the material behavior after long treatment by an etchant, an improvement on the loading device of the test setup and work on potential fracture mechanics specimen geometries. In the past year, modifications have been made to the STM to improve the performance of different components of the STM system. At the same time, an error analysis on the DIC has been conducted and a new approach to STM/DIC error analysis has been examined.			
14. SUBJECT TERMS			15. NUMBER OF PAGES 10
			16. PRICE CODE
17. SECURITY CLASSIFICATION OF REPORT UNCLASSIFIED	18. SECURITY CLASSIFICATION OF THIS PAGE UNCLASSIFIED	19. SECURITY CLASSIFICATION OF ABSTRACT UNCLASSIFIED	20. LIMITATION OF ABSTRACT UL

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**Final Report to the
Airforce Office for Scientific Research**

RECEIVED AUG 04 2003

Grant No. F49620-99-1-0091

***Determination of Strain Fields at the Nanometer Scale
with Applications to MEMS Designs and
Micromechanics***

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February 2002

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GRADUATE AERONAUTICAL LABORATORIES
CALIFORNIA INSTITUTE OF TECHNOLOGY



Progress Report
to the
Airforce Office of Scientific Research

DETERMINATION OF STRAIN FIELDS
AT THE NANOMETER SCALE WITH APPLICATIONS TO
MEMS DESIGN AND MICROMECHANICS

Research Grant Number:
F49620-99-1-0091

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Ioannis Chasiotis, Ying Huang²

Pasadena, December 1999

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Determination of Strain Fields at the Nanometer Scale with Applications to MEMS Design and Micromechanics

ABSTRACT

The following summarizes progress on the program made in the period of August 1998 until September 1999. Several aspects of the material behavior and characteristic were addressed such as the material constants, the surface condition and the material behavior after long treatment by an etchant, an improvement on the loading device of the test setup and work on potential fracture mechanics specimen geometries. In the past year, modifications have been made to the STM to improve the performance of different components of the STM system. At the same time, an error analysis on the DIC has been conducted, and a new approach to STM/DIC error analysis has been examined.

1. OBJECTIVE

This project is focused in the determination of mechanical properties of thin films manufactured by polycrystalline silicon using the surface micromachining method. In addition to the measurement of the mechanical properties by use of an Atomic Force Microscope (AFM) or a Scanning Tunneling Microscope (STM) with the aid of the Digital Image Correlation Method (DIC) several other aspects of the fracture behavior are addressed such as the effect of surface roughness, effects of notches to the fracture of the specimen in conjunction with grain size, etc. The STM, DIC have been re-evaluated to determine their capabilities and proceed with refinements on the instrument and the correlation method as well as identification of their limitations.

2. APPROACH

The main research focus was the experimentation with specimens manufactured by the Center of Microelectronics in North Carolina (MCNC) during MUMPs19 and MUMPs21 runs (figure 2). The test device is a miniature tensile tester designed in house (Figure 1) and the method that has been used so far to extract mechanical property values from experimental data utilizes a linear spring model to evaluate the experimental data. A thorough record of the specimens' condition before and after testing was conducted by means of an optical and an Atomic Force microscope. In this way, additional information such as grain size, undercut dimensions, surface roughness, etc. with respect to the etching time has been determined. A Finite Element Model (FEM) of the complete test structure developed for ABAQUS has been used to evaluate computationally the

experimental results and determine the contribution of the convergent sections of the paddle to the value of the total displacement (see figure 2). The tensile tester was initially designed to take advantage of the electrostatic forces that can be exerted between two conductive surfaces that are separated by a non-conductive medium. The method has been used for the data obtained so far but due to certain voltage-friction limitation, a new, more efficient gripping method has been developed that makes use of Ultraviolet (UV) adhesive. Electrostatic forces are applied in order to force the thin polysilicon film to lie flat on the substrate (due to the residual stresses in the released film it is bent up or down (figure 3,4)). It is then approached by a transparent glass grip (is chosen to be transparent so that the UV light will pass through it and cure the underlying glue layer) to the film surface (figure 5). Then the two surfaces of the substrate and the specimen paddle which electrostatically adhere, are repelled by charging both with the same charge so that the film is repelled from the substrate surface and adheres to the grip that is covered by a thin UV adhesive layer. A two-minute UV cure of the adhesive follows.

3. PROGRESS

1. The experimental results in conjunction with the finite element analysis of the specimen geometry provided an accurate value of the elastic modulus without any assumptions. The Young's modulus value was found as 165.9 ± 5.2 GPa and the value for the fracture strength as 0.96 ± 0.1 GPa. These values are in agreement with previous reports from other laboratories that applied different methods.
2. A diffraction analysis has been performed on the specimens and has shown two particular orientation patterns of the top surface of the specimens with (111) and (110) dominating diffraction planes. There is definitely an orientation preference in the texture but this is not very strong. This finding may be an explanation for the fact that the values of modulus are in the middle of the acceptable range (131GPa - 189GPa) of values for the specific crystallographic orientations and close to the elastic constant (168 GPa) in the [110] direction.
3. The value of the fracture strength for MUMPs21 is considerably higher than that measured for MUMPs19 in a previous work. It has also been identified that the low strength of specimens from MUMPs19 can be attributed to the long exposure time in hydrofluoric (HF) solution (to release them) when compared with the time used for the MUMPs21 specimens. The value of fracture strength for MUMPs21 specimens is lower than that reported by other researchers and can be directly attributed to the fact that the etching time was longer than that the others have reported. *Thus the effect of the HF release time on specimen quality is not negligible.* Further study, using an AFM, shows that the surface roughness and texture are a

strong function (figure 6) of specimen exposure to HF. An increased HF exposure results in increased non-uniform surface roughness and the appearance of deep depressions (figure 7) at individual areas, but not uniformly over the whole surface. Those grooves occur at the grain boundaries that are etched more readily and faster than the grains themselves. This implies that the knowledge of the actual surface roughness of the sample depends on parameters that are independent of the manufacturing process. It is the release process that reveals the grains and causes the deep etching of the grain boundaries.

The deep surface grooves act as micro-notches and can thus initiate fracture. This is probably the reason why the fracture strength is very low after long etching times. Observations have also shown that stiction phenomena can cause long-term problems to the specimens: this stiction facilitates HF encapsulation under the film so that the etching process can continue with slow rates. The surface roughness change under different exposure times in HF has been studied to the extent that measurements were possible.

4. We have improved the loading device with a preliminary gripping method. Breakdown voltage and charging problems that occur due to the nature of the applied electrostatics can be resolved by a new approach that is under development. Thus, electrostatics are not needed and there is no slip between the sample and the grip and no charging during the test due to the high voltage. The before-mentioned process eliminates the need of mechanically pressing the grip against the paddle and the substrate, and eliminates a potential pre-test specimen damage.
5. A study of two different notched specimen configurations has been preformed to investigate the necessary geometrical parameters to design and manufacture fracture mechanics specimens at MCNC. The dimensions of the notches were defined by the imposed minimum manufacturing requirements by MCNC. For the calculation of the design parameters an analytical notch theory was used as a first approximation. However a finite element model has also been constructed in order to capture the real strain\stress field on the surface of the specimen incorporating any end effects. The particular experiment has been designed to capture a possible effect of the grain structure (figure 8) of thin polycrystalline films on their fracture behavior. AFM data show an influence of the grain structure to the direction of crack propagation and a preference for intergranular fracture.
6. The STM is used to investigate material deformations at the submicron scale. It has a resolution ranging from below an angstrom to several tens of nanometers. However, problems had been experienced regarding the consistency of the resolution. In addressing these problems, modifications have been made on the STM scanning system, the acquisition system and the feedback control system.

When the STM was first built, the scanning was performed by one piezo-ceramic actuator. The in-plane motion was controlled by applying a voltage difference on the opposite electrodes of the piezo-ceramic tube, while the out-of-plane motion was actuated by applying a voltage difference on the inner and outer electrodes of the same tube. Consequently, there was a coupling problem between the in-plane and out-of-plane motions. To solve this problem, a new design was implemented, so that two piezo-ceramic tubes are used to actuate in-plane and out-of-plane motions separately. The inner tube, whose aspect ratio is more favorable for bending, controls the in-plane movements. It is fixed rigidly through two of ceramic connectors to the outer tube, which actuates the out-of-plane motion. Both of the piezo-actuators were assembled into the STM head. Scanning with the decoupled piezo-actuators showed that the scanning quality was improved.

Once the tunneling current is acquired, it is amplified and converted into voltage. This first stage amplification is accomplished inside the STM head. Experiments showed that considerable noise was introduced at this stage of amplification. The amplification circuits were assessed and a better circuit was designed. The new circuit incorporates three amplification stages instead of one and filters out noises from power supplies at each stage. Calibrations confirmed that the modified first stage amplification introduced far less noise than the old design.

The other crucial element of the STM system is the feedback control system, which keeps the tunneling current constant by feeding a voltage correction into the piezo-actuator controlling the out-of-plane motion. Each component of the STM control system is known, except for the piezo-ceramic actuator. In order to find out the dynamic behavior of the piezo-ceramic actuator, a frequency response test was conducted on the actuator and the first stage amplifier. Based on the dynamic response, the controller for the actuator and the amplifier was modeled via MATLAB. Both the step response and dynamic simulations for the analytical model controller showed that the current sampling frequency was set too fast for the system to respond. However, the controller was modeled linearly in, while the real one in the STM system is non-linear. Further investigations need to be carried out in this area.

7. The Digital Image Correlation (DIC) is used to extract the displacements and strains from the 3-D data of the undeformed and deformed surfaces. In the DIC, a minimum of the least square correlation coefficient gives the real in-plane displacements and strains. This method was extended to 3-D, and the out-of-plane displacement was included in the correlation coefficient. Problems arose from this. An error analysis of the program based on this correlation scheme was conducted. The scheme was evaluated, and a different method was proposed.

The current DIC program was evaluated by feeding in computer generated images with known strains and displacements. Different parameters for the correlation have been tried to

estimate the influence on the correlation result. High frequency noises with different amplitudes were added in to test the robustness of the program. Results showed that the DIC program tends to overestimate the strains. The size for the correlation subset plays an important role. Depending on the noise level, the noise is more negligible for larger deformations.

4. PERSONNEL INVOLVED INCLUDING POSITION AND MAN-YEARS

This project is supervised by Prof. W.G. Knauss of Aeronautics and Applied Mechanics and involves two graduate students; Ioannis Chasiotis and Ying Huang who are currently on the fourth and fifth year, respectively, of their graduate studies at the California Institute of Technology.

5. CONFERENCE PROCEEDINGS

There has been a conference proceedings publication:

- Ioannis Chasiotis, Wolfgang G. Knauss, "Investigation of Thin Film Mechanical Properties by Probe Microscopy", Proceedings of SPIE 3512, pp. 66-75, (1998)

6. PRESENTATIONS

The work has been presented at the following conferences – meetings:

- Ioannis Chasiotis, Wolfgang G. Knauss, "Investigation of Thin Film Mechanical Properties by Probe Microscopy", International Society for Optical Engineering Symposium, 21-23 September 1998, Santa Clara, CA
- Ioannis Chasiotis "Investigation of Thin Film Mechanical Properties by Probe Microscopy", 23 September 1998, Southern California American Vacuum Society Symposium, Orange CA
- Wolfgang G. Knauss, Ioannis Chasiotis, "Mechanical Properties of Thin Polysilicon Films by Means of Probe Microscopy" MRS Symposium, 2 December 1998, Boston, MA

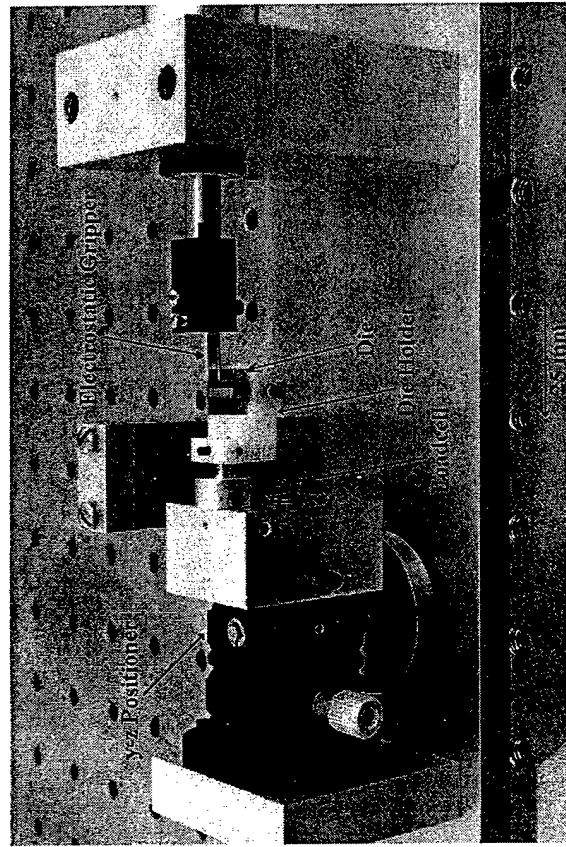


Fig. 1 Experimental Setup

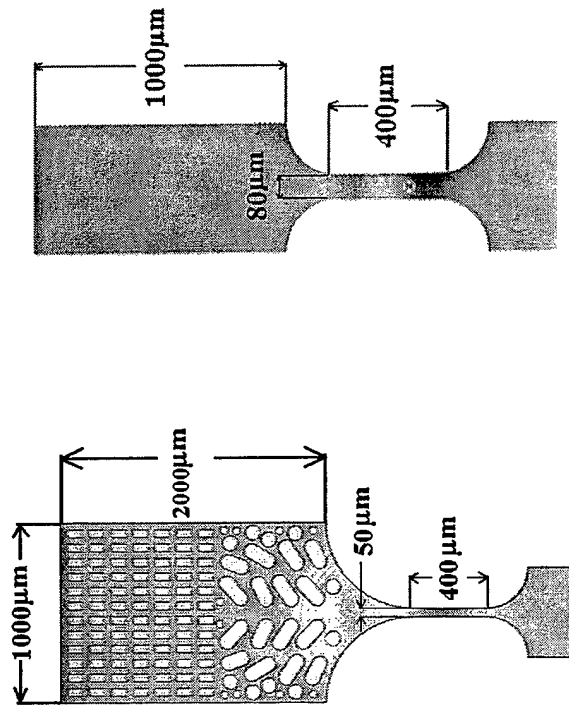


Fig. 2. Test Specimens

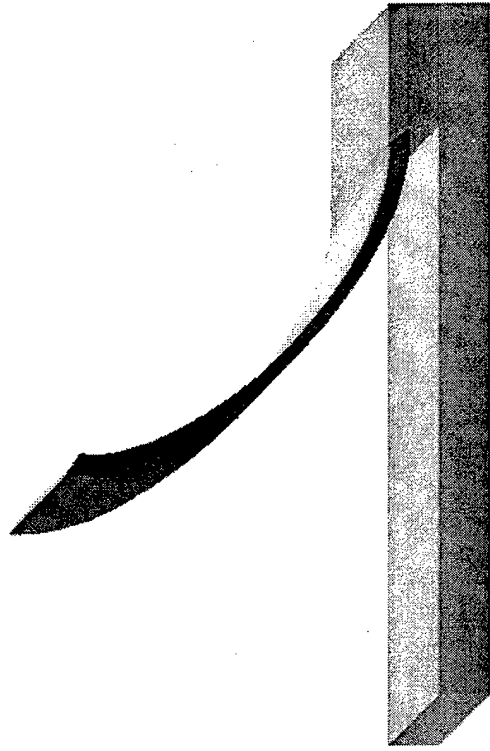


Fig. 3. Thin film curved up due to \pm stress gradient from interface to top surface

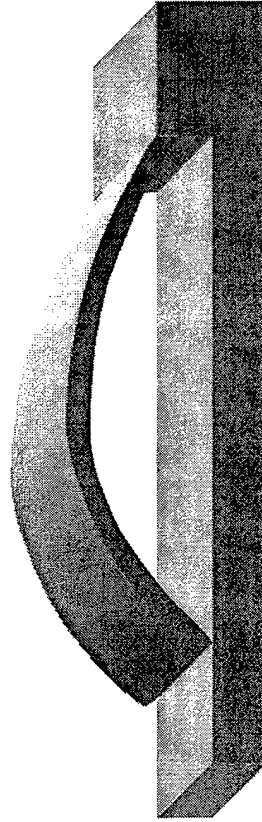
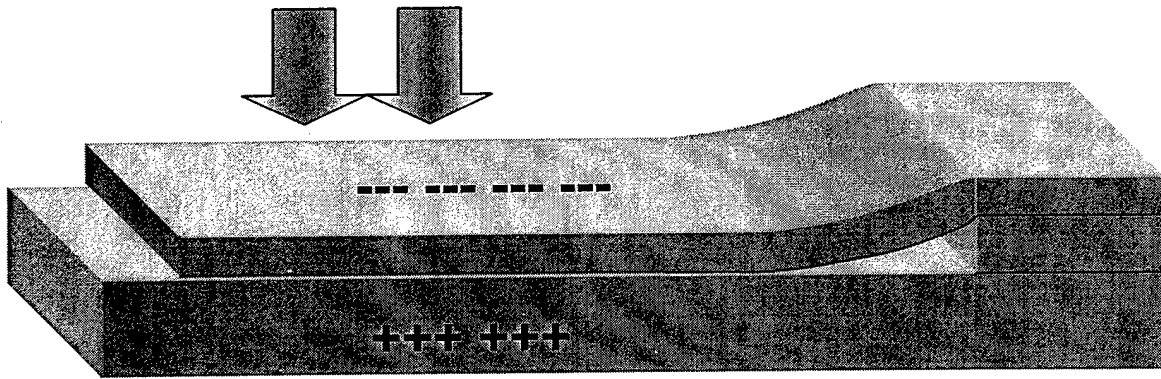
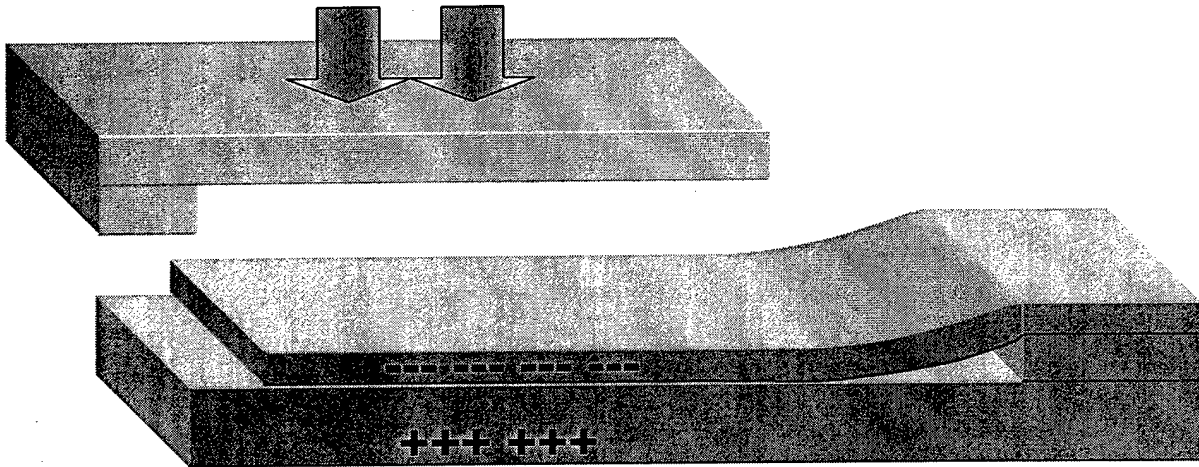


Fig. 4. Thin film curved down due to \pm stress gradient from interface to top surface

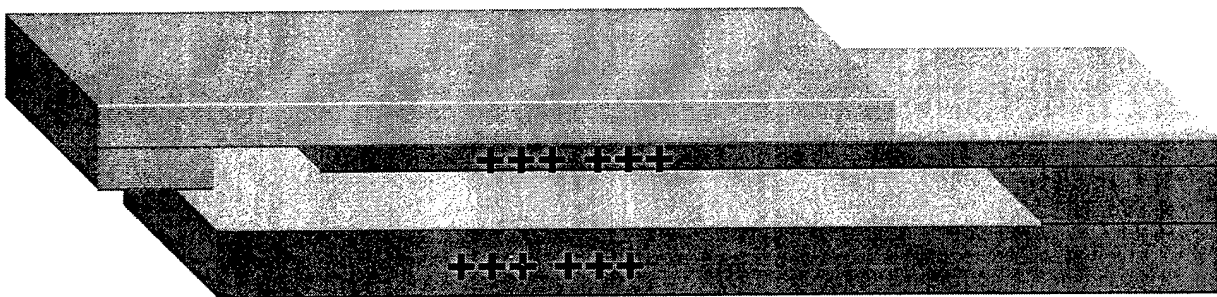
APPENDIX



A



B



C

Fig. 5. Successive steps in film gripping

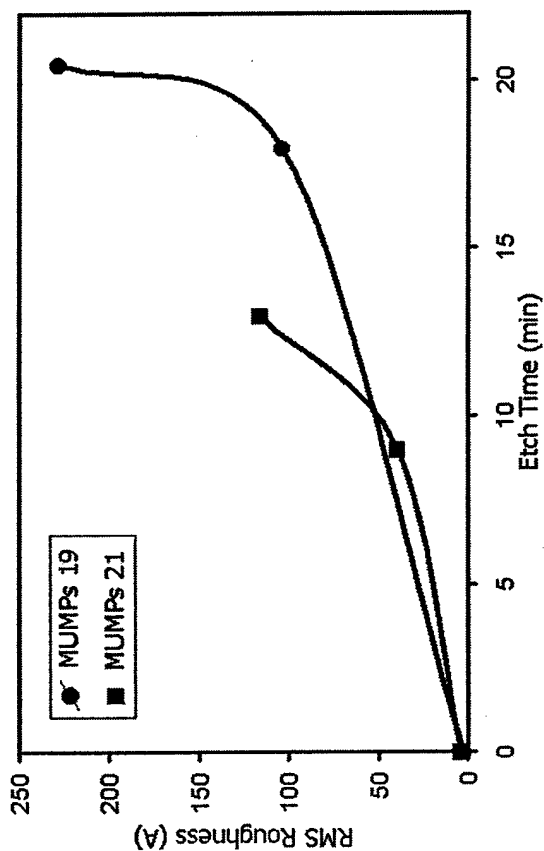


Fig. 6. Surface roughness as a function of etch time

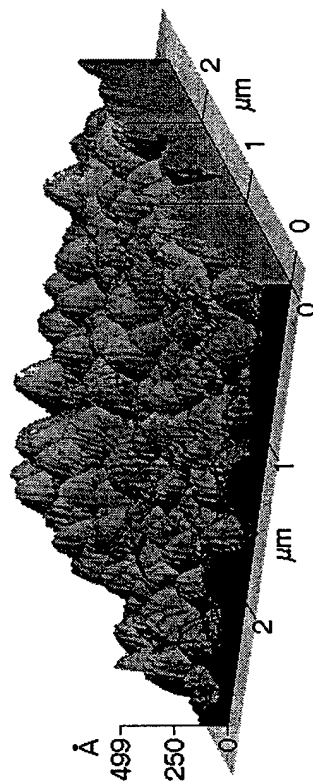


Fig. 7. Surface undulation due to HF etching

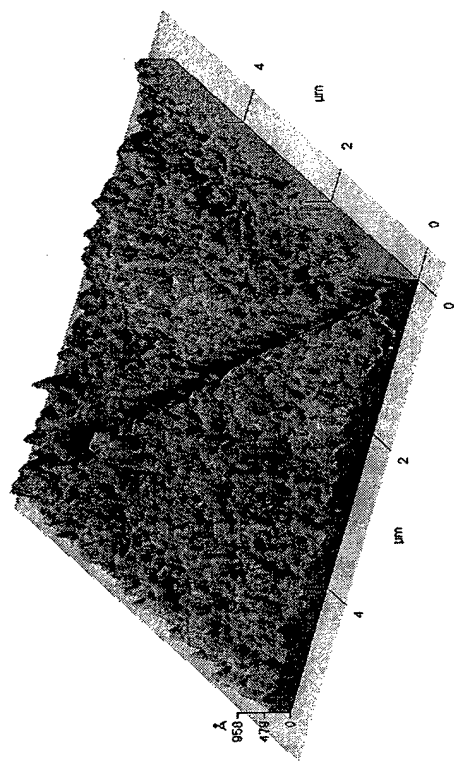


Fig. 8. Running crack in across a 2 micron film

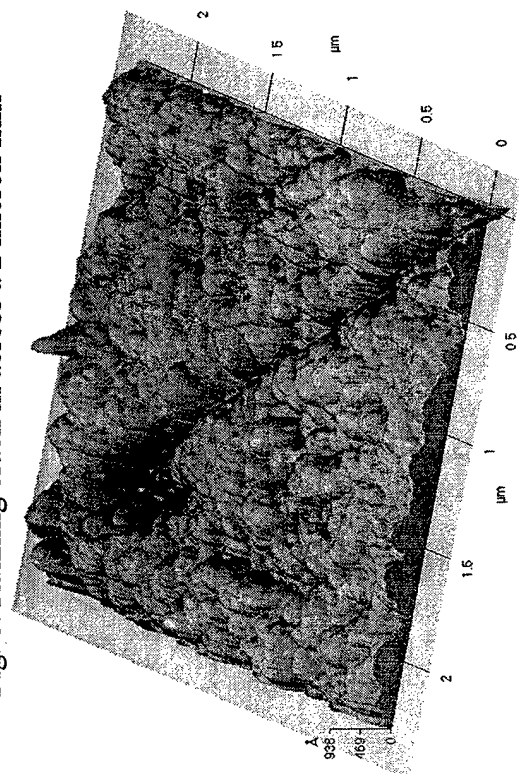


Fig. 9. Detail of the crack above and the crack arrest